CONVEYOR LUBRICANTS FOR USE IN THE FOOD AND BEVERAGE INDUSTRIES

Cross-Reference to Related U.S. Patent Applications

This application is a continuation-in-part of copending U.S. Patent Application Serial No. 10/294851 filed November 14, 2002, now allowed.

FIELD OF THE INVENTION

The present invention relates to lubricant concentrates and lubricant use compositions, and in particular to lubricant compositions which are employed as conveyor lubricants for use in the food and beverage industries, and to methods of using the same.

BACKGROUND OF THE INVENTION

In most packaging operations, including beverage operations, containers are moved along mechanized conveyor systems in an upright position from station to station wherein various operations including filling, labeling, sealing, capping, and the like are performed on the containers. During some of the operations, the containers are open. In particular in the beverage industry, it is important that the containers move without hindrance along the conveyor such that no liquid is spilled onto the conveyor. This is particularly important for dairy based beverages such as milk because milk can coagulate on the equipment surfaces.

Such conveyor systems are thus typically lubricated to reduce friction between the package and the load bearing surface of the conveyor. Thus, these lubricants are typically applied to the conveyor belts to reduce friction between the package and the conveyor which facilitates unhindered conveyance of bottles on the conveyor belt. These lubricants may also be referred to as chain conveyor or belt lubricants.

Not only are good lubricating properties important, there are other important considerations when selecting a lubricant for use in the beverage bottling industry. One such consideration is that the lubricant be compatible with the beverage such that it does not form coagulates or other solid deposits when it accidentally contacts spilled beverages on the conveyor system. The lubricant must also be readily cleaned from the equipment.

In the past, the lubricants commonly used on the load bearing surfaces of these conveyor systems typically contained fatty acid soaps as the active lubricating ingredient.

These fatty acid lubricants provided excellent lubricity, but are also known for forming insoluble precipitates in the presence of cations found in hard water such as calcium and magnesium. These precipitates can cause clogging of nozzles and subsequent loss of lubricant flow to conveyor surfaces. Water softening agents and chemical chelating or sequestering agents, such as EDTA, can be employed with these lubricants to prevent formation of such precipitates. However, strong chelating agents such as EDTA are also known for leaching calcium out of concrete which causes pitting in the concrete floor surfaces which are commonly found in beverage plants and dairy operations.

Amine-based lubricants are known and may be used as an alternative lubricant to the fatty acid based lubricants for lubricating conveyor systems. However, amine based lubricants tend to form precipitates with polyvalent anions such as carbonates and sulfates which are also present in hard water. This can lead to clouding of the lubricant solution. Furthermore, amine-based lubricants have been known to cause coagulation of milk which is a disadvantage for use in dairy conveyor operations. The coagulation of milk results in soil build-up on equipment and environmental surfaces. This soil is aesthetically unappealing, difficult to clean and may, for a thorough cleaning, require dismantling of the equipment.

Soil build-up can also cause fouling of moving parts on container filling equipment. Such fouling can become a harbor for bacterial growth and subsequently lead to problems with product quality. More frequent cleaning and longer cleaning times may be required.

Preventing soil build-up resulting from milk coagulation can reduce the amount of time required for cleaning. Dairy operations and beverage plants are typically cleaned once a day providing that no problems arise from interaction between a lubricant solution contacting spilled products.

Consequently, there remains a need in the art for an improved lubricant that not only exhibits excellent lubricity, but which also functions well in hard water, is non-corrosive to soft metals, steel and concrete, and which is compatible with and does not coagulate beverage products such as milk.

SUMMARY OF THE INVENITON

The present invention relates to lubricant concentrates and diluted lubricant use compositions which include an effective lubricating amount of at least one ether carboxylate and at least one defoamer/surfactant.

Suitably, the ether carboxylates employed herein have from about 8 to 20 carbon atoms, and more suitably about 12 to 18 carbon atoms. In one embodiment, the ether carboxylate is an oleyl ether carboxylate having from about 16 to 18 carbon atoms.

The ether carboxylates suitably have alkoxylation in the range of about 30 to about 20 moles, more suitably about 5 to about 15 moles of alkoxylation. The ether carboxylates may be ethoxylated, propoxylated or both.

One specific example of an ether carboxylate suitable for use herein is that sold under the trade name of EMULSOGEN® COL 100 available from Clariant Corp. based in Basel, Switzerland, an oleyl ether carboxylate having 16-18 carbon atoms and 10 moles of ethoxylation.

The ether carboxylate finds utility and exhibits lubricity at concentrations of about 0.1 to about 75 wt-%, suitably about 0.25 wt-% to about 50 wt-%, more suitably about 0.5 wt-% to about 15 wt of the lubricant concentrate.

Of course, the lubricant concentrates according to the invention may be diluted with water at a ratio of about 1 to about 1000 water to lubricant concentrate. For some applications, diluted use concentrations of the lubricant compositions in water may be in the range of about 0.1 wt-% to about 10 wt-% of the concentrate in water, and more suitably 0.25 wt-% to about 5 wt-% of the lubricant concentrate in water. For some applications, it is desirable to employ diluted use compositions at concentrations of greater than about 0.3 wt-%, more suitably greater than 0.4 wt-% and most suitably greater than about 0.5 wt-%, and up to about 10 wt-%, although these ranges may be varied depending on the application for which the lubricant is being used.

The ether carboxylate may be advantageously employed in combination with a foam suppression additive. One example of such an additive is an alkoxylated alcohol. Suitably, the alkoxylated alcohols have about 8 to 16 carbon atoms and more suitably the alkoxylated alcohols have about 11 to 13 carbon atoms. In one embodiment, the alkoxylated alcohol is a propoxylated alcohol having about 9 to 11 carbon atoms, a commercially available example of which is DEGRESSAL® SD-20 available from BASF Corp. in Mount Olive, New Jersey.

Other optional ingredients may be employed in the lubricant compositions according to the invention. The ether carboxylate lubricants may be employed in combination with anti-corrosion agents such as ether diamines and/or dicarboxylic acids or salts thereof. Hard water, particularly well water, has been found to be highly corrosive to mild steel, for example.

Other adjuvants may also be optionally employed in the lubricant compositions according to the present invention. Such adjuvants include, but are not limited to, viscosity modifiers, soil anti-redeposition agents, preservatives, dyes, fragrances, anti-foaming agents, soil suspension and solubilizing agents, penetrants, antimicrobial agents, other surfactants, other hydrotropes, and so forth.

The lubricating compositions according to the present invention exhibit excellent lubricating properties, particularly in hard water conditions such as with well water, are noncorrosive, are non-pitting to both soft metals, steel and concrete, and do not coagulate dairy products such as milk. This superior combination of properties makes the inventive lubricants highly desirable for use as lubricants in food and beverage operations.

The lubricating compositions according to the present invention are advantageously employed as conveyor lubricants in food and beverage operations. They have been found to be particularly useful as dairy conveyor lubricants because, unlike prior lubricants, they do not cause coagulation of milk.

The lubricating compositions according to the present invention are especially suited for the high pressure lubricity requirements of dairy plant cooler areas. Dairy plant conveyor systems, in addition to the relatively low pressure areas of the production lines where individual product packages are conveyed, have in-floor conveyors sections in the cooler areas where cases of milk are stacked six high on the conveyor tracks. These stacks can weigh as much as two to three hundred pounds each. At any one time as many as thirty stacks of cases can be in transit on one conveyor section. The high pressure lubricity properties of the present invention, as demonstrated by the test results using the Falex high pressure lubricity test system, are especially suited for the lubricity requirements of in-floor conveyor applications found in dairy plant cooler areas.

The lubricating compositions according to the present invention are typically prepared as concentrates, and then diluted to an end use concentration prior to use.

The present invention further relates to methods of using the lubricant compositions according to the present invention. One such method includes lubricating a continuously-moving conveyor system for transporting packages wherein the conveyor system is wetted with an aqueous lubricant composition including at least one ether carboxylate and at least one foam destabilizer according to the present invention.

The lubricant composition may be provided to the end user as a concentrate, or the method may include the step of diluting the concentrate prior to application of the concentrate to the desirable location of the conveyor system. The lubricant composition may be applied to the conveyor system using a spray method. Application may involve applying the lubricant to the package itself.

These and other advantages of the present invention will be more readily understood by those skilled in the art from a reading of the following detailed description.

DETAILED DESCRIPTIONS OF THE PREFERRED EMBODIMENTS

While this invention may be embodied in many different forms, there are described in detail herein specific embodiments of the invention. This description is an exemplification of the principles of the invention and is not intended to limit the invention to the particular embodiments illustrated.

The present invention relates generally to a lubricant concentrate, diluted use lubricant compositions and to methods of using the same.

A. *The Ether Carboxylate Lubricants*

The lubricant compositions of the present invention include at least one ether carboxylate. The ether carboxylates suitably have the following general formula:

$$R\text{-}(OCH_2CH_2)_n\text{-}OCH_2COO\text{-}X$$

where X is an alkali metal, amine, alkanolamine, ether diamine, ammonium salt or H (free acid), $R = C_8 - C_{20}$ and n is about 6 to about 18, more suitably $R = C_{12} - C_{18}$ and most suitably $R = C_{16} - C_{18}$. In one embodiment, an oleyl ether carboxylate is employed in the lubricant composition. Longer alkyl chains of about 16-18 carbon atoms have been found to provide excellent solubility and lubricity. Ether carboxylates with this structure have excellent hard water tolerance and lime soap dispersing properties. These

properties contribute to keep the conveyor lines clean. The ether carboxylates according to the invention may be propoxylated, ethoxylated, or both. Ethoxylation may range from about 3 to about 20 moles ethylene or propylene oxide or mixtures thereof, and more suitably about 5 to about 15 moles ethoxylation. In one embodiment, the ether carboxylate has about 10 moles of ethoxylation.

In some embodiments, the ether carboxylates are propoxylated. The amount of propoxylation may range from about 3 to about 20 moles propoxylation and more suitably about 2 to about 10 moles propoxylation.

In other embodiments, both ethylene oxide and propylene oxide may be employed. The resultant ether carboxylates may have about 3 to about 20 moles ethoxylation and about 3 to about 20 moles propoxylation, and more suitably about 5 to about 15 moles ethoxylation with about 2 to about 10 moles propoxylation.

The ether carboxylates described herein have been found to have excellent hard water tolerance and lime soap dispersing properties.

An example of a commercially available ether carboxylate is EMULSOGEN® COL 100 available from Clariant Corp. based in Basel, Switzerland. This is an oleyl ether carboxylate having 16-18 carbon atoms and 10 moles of ethoxylation. Ethoxylation improves solubility of the lubricant.

The concentrates are typically diluted with water in the range of about 1 to about 1000, 1 to about 500 and more suitably about 1 to about 200.

The ether carboxylate finds utility and exhibits lubricity at concentrations of about 0.1 to about 75 wt-%, suitably about 0.25 wt-% to about 50 wt-%, more suitably 0.50 wt-% to about 15 wt-%.

For some applications, the lubricant concentrates may be in the range of about 1 wt-% to about 10 wt-%.

The ether carboxylate lubricants provide excellent high pressure lubricity. High pressure lubricity can be measured using a Falex testing instrument.

B. *Foam Suppression Additives*

A foam suppression additive or foam destabilizer is suitably employed in combination with the ether carboxylate lubricant.

Suitable foam destabilizers include those that fall under the general category of non-ionic surfactants. One class of suitable non-ionic surfactants includes the alkoxylated alcohols including ethoxylated and propoxylated alcohols. Suitably, the

alkoxylated alcohol has about 8 to about 16 carbon atoms, more suitably about 9 to 11 carbon atoms.

A specific example of a suitable alkoxylated alcohol includes, but is not limited to, DEGRESSAL® SD 20, a propoxylated alcohol having a molecular weight of 1320 g/mole available from BASF Corp. in Mount Olive, NJ. Ethoxylated alcohols are commercially available from the Dow Chemical Co. in Midland, MI under the tradename of TERGITOL®.

C. *Corrosion Inhibitors*

The ether carboxylate lubricants of the present invention may be advantageously used in combination with corrosion inhibitors. Examples of such corrosion inhibitors include, for example, ether amines, polycarboxylic acids such as carboxylic diacids, triacids, as well as the phosphate esters, or other salts, including sodium or potassium salts, thereof, phosphonated amine oxides, and so forth.

The present inventors have found that when at least one ether carboxylate lubricant is employed in combination with at least one ether amine and/or at least one polycarboxylic acid or salt thereof, and preferably both, the lubricant composition prevents corrosion of mild steel. For stainless steel, anti corrosion agents, in particular the dicarboxylic acid, provide no further benefits.

The ether amines suitable for use herein include linear and branched, and saturated and unsaturated alkyl ether amine compounds.

Suitable ether amines and diamines include those having the following general formulas:

$$R_1—O—R_2—NH_2$$

and

$$R_1—O—R_2—NH—R_3—NH_2$$

and mixtures thereof, wherein R_1 may be linear C_6-C_{18}, R_2 may be a linear or branched C_1-C_8 alkyl, and R_3 is a linear or branched C_1-C_8 alkyl group. Ether diamines such as these are described in commonly assigned US 5723418 and US 5932526, and in US 6306816, each of which is incorporated by reference herein in its entirety.

More specific examples of suitable ether diamine anti-corrosion agents are those having the following general formula:

$$ROC_3H_6NHC_3H_6NH_2$$

where R is a straight or branched chain alkyl group having from about 8 to about 30 carbon atoms. Examples of such ether diamines include, but are not limited to, octyl/decyloxypropyl-1,3-diaminopropane; isodecyloxypropyl-1,3-diaminopropane; isododecyloxypropyl-1,3-diaminopropane available from Tomah Products, Inc. under the tradename DA-16; dodecyl/tetradecyloxypropyl-1,3-diaminopropane; isotridecyloxypropyl-1,3-diaminopropane available from Tomah Products, Inc. under the tradename DA-17; tetradecyloxypropyl-1,3-diaminopropane available from Tomah Products, Inc. under the tradename of DA-18; and so forth; to mention only a few.

Another specific example of a commercially available ether amine is TOMAH® DA1618 which is a mixture of 60% N-dodecyloxypropyl-1,3-diaminopropane and N-tetradecyloxypropyl-1,3-diaminopropane available from Tomah Products, Inc. and having the following general formula:

$$ROCH_2CH_2CH_2NHCH_2CH_2CH_2NH_2$$

where R is an alkyl group having 12 and 14 carbon atoms.

Suitable examples of polycarboxylic acids or salts thereof are those having the following general formula:

$$HOOC\text{-}R\text{-}COOH$$

where R is an alkyl group having from about 1 to about 8 carbon atoms and more suitably about 1 to about 4 carbon atoms.

In some embodiments, the corrosion inhibitors are polycarboxylic acids such as dicarboxylic acids. Examples of useful dicarboxylic acids include, but are not limited to, adipic acid, glutaric acid, succinic acid or mixtures thereof. In one embodiment, a mixture of adipic acid, glutaric acid and succinic acid

The corrosion inhibitors are useful at concentrations of about 0.05% to about 25% and more suitably about 0.1% to about 20% in the concentrate. In one embodiment, the concentration of the corrosion inhibitor is about 0.5 wt-% to about 3 wt-%.

One example of a useful corrosion inhibitor is SOKALON® DCS diacid mixture available from BASF, Inc.

In one embodiment, an ether diamine is employed in combination with a mixture of diacids and a phosphonated amine oxide.

D. Other Surfactants

Other surfactants may be optionally employed in the lubricant concentrates and diluted-use compositions of the present invention. Such surfactants should be selected and employed in amounts such that the properties of the lubricant according to the present invention, such as the non-coagulation of milk, for example, are not negatively impacted. Such surfactants are known to those of ordinary skill in the art.

E. Hydrotropes

Other hydrotropes may be optionally employed in effective amounts in the lubricant concentrates and diluted-use compositions according to the present invention to provide viscosity control and cold temperature stability of the concentrate. Examples of optional hydrotropes include, but are not limited to, the alkali salts of aromatic sulfonates including sodium linear alkyl naphthalene sulfonate, potassium linear alkyl naphthalene sulfonate, sodium xylene sulfonate, potassium xylene sulfonate, potassium or sodium toluene sulfonate, potassium or sodium cumene sulfonate, and so forth; n-octenyl succinic anhydride (NOS); ammonium cumene sulfonate; alkyl polyglucoside; and so forth. The above list is intended for illustrative purposes only and is not exhaustive. Hydrotropes are known to those of skill in the art and there are numerous types available for use.

E. *Antimicrobial Agents*

It may be desirable to improve antimicrobial efficacy by adding, in addition to the other ingredients, one or more antimicrobial agents. Generally, any solid or liquid chemical agent having microbiocidal efficacy may be used in the invention.

Chemical compositions known to impart microbiocidal efficacy include iodophors, phenolics, quaternary ammonium compounds, and so forth.

More specific examples of antimicrobial agents include, but are not limited to, cationic surfactants such as alkyl and benzyl quaternary compounds like N-alkyl (C_{12-18}) dimethylbenzyl ammonium chloride, N-alkyl (C_{14-18}) dimethylbenzyl ammonium chloride, N-tetradecyldimethylbenzyl ammonium chloride monohydrate, dimethyl didecyl ammonium chloride, and N-alkyl (C_{12-14}) dimethyl 1-napthylmethyl ammonium chloride which are available commercially from manufacturers such as Stepan Chemical Company, and so forth. The above lists are not exhaustive and are intended for illustrative purposes only. One of ordinary skill in the art has knowledge of such antimicrobial agents.

When present, an antimicrobial agent must have a concentration effectively necessary for the required action to be provided. Generally, in the lubricant concentrate the concentration of antimicrobial agent may range from about 0.1 to 10 wt-%, preferably from about 1 to 8 wt-%, and most preferably from about 2 to 6 wt-%.

F. *Other Adjuvants*

The lubricant concentrate and, in turn, lubricant use-composition of the invention may also include one or more other adjuvants to modify the character or properties of the compositions. Examples of other commonly employed adjuvants include viscosity modifiers, soil anti-redeposition agents, preservatives, dyes, fragrances, anti-foaming agents, soil suspension and solubilizing agents, as well as penetrants, and so forth. One of ordinary skill in the art is well versed in the type of adjuvants employed in such lubricant compositions.

The lubricant use-compositions of the present invention may be formulated as concentrates, and then later diluted to the lubricant use-composition, the dilution depending on the application for which the lubricant use-composition is being employed. Generally, the lubricant concentrate may be diluted with water anywhere from about 1 to about 1,000 times, and more suitably about 1 to about 400, and even more suitably about 1 to about 200 times, to provide the lubricant use-dilution which is desirable.

The lubricants according to the present invention function excellently in hard water, and thus require no chelating or sequestering agents to prevent precipitates

from forming in hard water environments. The ether carboxylate based lubricant according to the present invention has been found to provide water hardness compatibility for the lubricant solution with water containing as much as 20 grains per gallon of water hardness without any chelating or sequestering agents. This is beneficial because strong chelating agents such as EDTA, for example, have been found to remove calcium and other cations from concrete leading to pitting of the concrete. Thus, being free from strong chelating or sequestering agents is also an added advantage.

Another advantage of using the ether carboxylate based lubricants according to the present invention is that they do not cause coagulation of dairy products, particularly milk.

The lubricant compositions according to the present invention may be employed as conveyor lubricants for conveyor systems which move product or packages along the conveyor system through a series of operations which are performed on each package. These lubricants are sometimes referred to in the industry as belt lubricants. The lubricant compositions of the present invention find particular utility in conveyor beverage and food operations, particularly bottling type operations, wherein packages or bottles are moved via a conveyor through a series of operations including filling, capping, and so forth. Such packages or bottles are typically open during some of the operations and are thus moved along the conveyor while open.

The lubricants may be provided to the end user as a concentrate which requires dilution according to a set of instructions to a diluted use composition, or the lubricants may be provided in an already diluted, ready-to-use form.

The lubricants may be applied to the desirable location using a variety of application methods. One typical method involves spraying the diluted use lubricant composition onto the conveyor system. This may involve a series of spray heads located along the conveyor system.

The lubricating compositions facilitate the unhindered movement of containers along the conveyor system. This is particularly important in beverage operations such as bottling operations, particularly for the dairy industry. Spilled beverages can result in having to shut down the operation for cleaning, thus, the unhindered movement of the bottles along the conveyor system is very important in these operations. Furthermore, it is important that the lubricant be compatible with the spilled beverage such that precipitation, coagulation and solid deposits do not form. The

lubricants of the present invention find particular utility in dairy operations because they do not cause coagulation of milk when they come into contact with it.

The lubricant compositions of the present invention may also be applied to the packages which are to be moved along the conveyor, although this is typically a less desirable method.

The following non-limiting examples further illustrate embodiments of the present invention.

EXAMPLES

TEST METHODS

1. Milk Coagulation

Three stainless steel panels, 3 inch by 5 inch each, were cleaned with EXPRESS® detergent and a sponge ringed with deionized water, acetone and then allowed to dry at room temperature.

2.5 grams of a 0.5% solution, which was prepared with 19 grain per gallon hardness well water, was pipetted onto a clean 3 inch by 5 inch stainless steel panel at room temperature. Two drops of commercial 2% milk, obtained from a local convenience store, was pipetted onto the center of the 0.5% use solution pooled on the panel. The drops of 2% milk and the 0.5% use solution were allowed to react for five minutes and then poured off the panel. The panel was allowed to dry at room temperature for several minutes and was then checked for appearance. Any sign of residue from coagulation of the milk, caused by reaction with the use solution, was recorded and photographs were taken.

Failed formulas left a ring of residue around the center where the milk droplet was placed and particles were visible on the panel surface.

Formulas that passed the test did not leave any residue of particles on the panel indicating that the milk did not precipitate from solution.

2. Milk Coagulation

Approximately 500 g (497.5 g) of 0.5% use solution which was diluted with 22 grain per gallon hardness well water was placed in a 600 milliliter beaker. One gram of 2% milk was pipetted into the beaker. The combined solution was covered and left to stand at room temperature for 12 minutes, one hour and in some cases 24 hours.

The solutions were observed after 12 minutes. Failed solutions showed precipitated particles on the beaker bottom, and in some cases, particles were floating in solution.

Formulas which passed did not cause precipitation of the milk but merely diluted it and formed a solution with the milk.

3. Slider Lubricity Test (DELRIN®; 0.5% in well water; midpoint/bandwidth)

Soft water with $NaHCO_3$ was used to prepare 100 mls of each test lubricant at the target concentration of 0.1 wt-% active amine compound. Soft water or a standard lubricant (0.1% Lubri Klenz® LF available from Ecolab, Inc. in St. Paul, MN) may be employed as the control.

The coefficient of friction (COF) for this composition was found to be 1.00 for mild steel on stainless. The formula was tested at 0.1 wt-% in distilled water containing 200 PPM $NaHCO_3$.

The DELRIN®, a thermoplastic prepared from acetal resin slider plate or a polished stainless steel slider plate (20.5 cm in diameter) was cleaned with distilled water and IPA. The chart recorder was turned on for 30 minutes and allowed to warm-up before calibration.

A 50 gram weight was added to the load cell and allowed to hang over the edge of the support. If the pen for the chart recorder did not record at 50, it was adjusted to 50 using the zero control. The weight was then removed and the zero mark adjusted. The pen input was set to 1 Volt and the chart speed set to 1 cm/min.

The plate was connected to an electric motor and the Slider was turned on such that the plate rotated at a steady rate. Solution was applied along the perimeter of the plate. A control was always tested first. To insure proper operation of the instrument and also to insure consistency, a maximum of three experimental lubricants were tested in between each control.

The appropriate rider piece (glass (189 g), mild steel (228 g) or polyethylene terephthalate (PET) disk) was attached to a load cell and placed on the plate in the are wetted by the lubricant solution. When the electric motor was switched on, the disk glided freely on the plate. The Slider plate was allowed to run for 5 minutes or until the force was level. The solution was refreshed 2-3 times during the run. The drag between the glass or mild stainless steel disk and the stainless steel plate was detected by the load cell. The output of the load cell was sent through signal conditioners to the chart recorder.

Various rider pieces may be tested with each lubricant if pertinent. Each lubricant and each rider were noted. The Slider plate was cleaned with distilled water and IPA after each lubricant.

The control may be run before and after each run, and should not be run any less than every 3 runs. The mid-point on the chart recorder relating to net force registered on the load cell should be recorded for comparative lubricity values.

The force between the glass, mild steel or PET rider piece and the stainless steel plate is detected by the load cell and transferred to a chart recorder. The value obtained from the control lubricant before and after the test lubricants is arbitrarily assigned a coefficient of friction (COF) of 1.00. Each trial run is then referenced to the control run, thus resulting in reports of relative COF. The lower the COF value, the better the lubricity. The relative COF was determined using the average force (midpoint of the data collected on the chart recorder) in the following formula:

$$\text{Force}_{S} / ((\text{Force}_{C2} - \text{Force}_{C1}) / \text{Run Order}_{C2}) * \text{Run order}_{S} + \text{Force}_{C1})$$

Where S = sample; C1 = control run before the test lubricants; and C2 = control run after the test lubricants. The run order for each sample is assigned such that the first test lubricant is 1, the second test lubricant is 2, and the third test lubricant is 3. The run order for C2 is n + 1, where n = the total number of samples tested between C1 and C2.

The COF for each lubricant is reported relative to the COF of the control. The lower the COF, the better the lubricity.

Alternatively, a standard may be run before and after each test run. The midpoint value which is equal to the net force from the load cell is recorded from the chart recorder readout, and the midpoint force value of the test sample is compared to the midpoint force value of the standard which is obtained from the chart recorder.

To validate the Slider results, the lubricant can be tested on a short section of a conveyor track with similar control parameters.

4. <u>Slider Lubricity Test (Mild steel on stainless steel/0.5% in soft water; midpoint/bandwidth)</u>

The same procedure is followed as in Test 3.

5. <u>Recirculated Lubricant Solution Falex Lubricity Test (ASTM D-2670)/Mild Steel and Soft Water</u>

A Falex Pin & Vee Block Test Machine for friction and wear testing available from Falex Corp. in Sugar Grove, IL was used to test lubricity. The pins and Vee blocks used in this testing were obtained from the Falex Corporation. The pins and Vee blocks were composed of AISI/SEA 3135 steel. Two liters of test lubricant solution was prepared in a 4 liter beaker using Soft water with 0.4 grains per gallon of hardness and with hard water having 19 grains per gallon of hardness. The test lubricant was weighed to the nearest 0.01g.

6. <u>Corrosion Test (Well water immersion)</u>

1100g of a 0.5% solution was prepared using water from the Golden Guernsey Dairy of Waukesha, WI (19 grain per gallon hardness as calcium carbonate or 325 parts per million as calcium carbonate). 500g of each solution was placed in a 600 ml beaker. Q-panels, 3 x 5 inch cold rolled steel corrosion test panels (cold rolled steel; low carbon SAE 1010; 1/4 hard - (Rockwell B65 to 70); ASTM A-366:QQS -698) from the Q-Panel company of Cleveland, Ohio) were added to each test solution. A paraffin cover was placed over each beaker to prevent evaporation. The panels were left in the test solution for 24 hours. The time may be varied as desired and as considered adequate for the application. The tests were conducted at room temperature. At the end of the tests the panels were removed and allowed to air dry. Once the panels were dry, they were rated for rust, i.e. corrosion, by visual inspection and the amount of rust was estimated.

7. <u>Foam Level Testing</u>

Foam level testing for dairy lube foam level reduction was conducted by preparing a five gram mixture of 4.75 grams of the various formulas with the addition of 0.25 gram of a foam destabilizer. A 200 gram solution at 0.5 wt-% for each formula was prepared in water of 19 grain per gallon hardness. This solution was added to a 500 milliliter stoppered graduated cylinder. The graduated cylinder with the solution was then inverted ten times. The standard product was tested at 0.5%, as produced, with no additives for foam destabilization.

The initial foam height was recorded, a timer was started and the foam level in the cylinder was recorded as a function of time.

Formulas which ,in comparison to the standard product, showed reduced foam height and a decrease in foam height as a function of time were then chosen for further foam level testing with the foam destabilizer at various concentrations and in combination with other selected foam destabilizers. A 200 gram solution at 0.5 wt-%

for each formula was prepared in water of 19 grain per gallon hardness. This solution was added to a 500 milliliter stoppered graduated cylinder. The graduated cylinder with the solution was then inverted ten times.

The initial foam height was recorded, a timer was started and the foam level in the cylinder was recorded as a function of time.

8. Foam level testing was conducted on a short section of conveyor track. A 0.5 wt-% solution of each formula in 14 grain per gallon well water was prepared, and each solution was then run on a ten foot long section of stainless steel conveyor track with no bottles for 3 ½ hours. The 0.5 wt-% solution was sprayed onto the track at a rate of 0.8 gallons per hour through three nozzles. The total application rate was 2.4 gallons per hour. The foam level was observed and recorded.

Example 1

The following lubricant concentrate was prepared.

Example 1

Table 1

Raw Material	Tradename	Function	Wt-%
Oleyl ether carboxylate, 10 moles ethoxylation	EMULSOGEN® COL 100	Lubricant	7.5
Sodium alkyl naphthalene Sulfonate; 50% active	PETRO® LBA	Coupling agent	7.0
C_9-C_{11} Alkoxylated alcohol	DEGRESSAL® SD 20	Defoamer/surfactant	5.0
Chloralyl triaza azoniaadamentane	DOWICIL® 75	Biocide/preservative	0.13
Dodecyl/tetradecyloxypropyl-1,3-diaminopropane	TOMAH® DA 1618	Corrosion inhibitor	2.85
Phosphated amine oxide	BURCOTERGE® PAO-35	Corrosion inhibitor	0.50
Dicarboxylic acid mixture	SOKALON® DCS	Corrosion inhibitor	0.85
Sodium gluconate, granular	Sodium Gluconate FCC/USP	Chelates iron; rust inhibitor	2.00
Water, zeolite softened	Soft water		73.41
Sodium hydroxide; 50%	NaOH 50%	pH adjuster	0.76

Comparative Example A

Table 2

Chemical description	Tradename	Function	A	B	C	D
Sulfo methyl ester	ALPHA-STEP MC-38	Lubricant	----	27.00	27.0	
Alkyl dimethyl benzyl ammonium chloride, 50%	Barquat® MB-50	Antibacterial	----	----	----	12.0
Sodium xylene sulfonate	Naxonate® 4L	Coupling agent	----		12.50	-
Hexylene glycol	Hexylene Glycol	Coupling agent	----	----	----	8.00
Tetra sodium ethylenediamine tetracetic acid powder 4 H_2O	Versene 220	Chelation	----	----	----	4.60
Linear alcohol 60-70%, 7 ethoxy	Surfonic® L24-7	Surfactant	----	----	----	1.0
Polyethylene glycol 15 cocamine	Varonic K-215	Lubricant	----	----	----	4.00
Coconut fatty acid	Kortacid® C70	Lubricant	----	----	----	11.25
Tall oil fatty acid	Unitol DSR 90	Lubricant	----	----	----	2.25
Triethanolamine, 99%	TEA 99%	Lubricant (salt additive)	----	----	----	12.00
Sodium alkyl naphthalene sulfonate; 50%	PETRO® LBA	Coupling agent	7.00	7.00	----	----
Dodecyl/tetradecyloxypropyl-1,3-diaminopropane	TOMAH® DA 1618	Corrosion inhibitor	2.85	2.85	2.50	
Phosphated amine oxide	BURCOTERGE® PAO-35	Corrosion inhibitor	0.50	0.50	----	
Dicarboxylic acid mixture	SOKALON® DCS	Corrosion inhibitor	0.85	0.85	0.85	
Sodium gluconate granular	Sodium Gluconate FCC/USP	Chelates iron; rust inhibitor	2.00	2.00	----	
Sodium laureth-13-carboxylate	SANDOPAN® LS 24N	surfactant	13.98	2.85	2.85	
Chloralyl triaza azoniaadamentane	DOWICIL® 75	Biocide/preservative	0.20	0.20	----	
Potassium hydroxide, 45% L	KOH 45%	pH adjuster	----	----	----	5.25
Water, zeolite softened	Soft water	Solution base	72.62	56.75	54.30	39.65

TOMAH® DA 1618 is an ether diamine available from Tomah Products, Inc. in Milton, WI.
SOKALON® DCS is a mixture of succinic, glutaric and adipic acids available from BASF Corp. in Mount Olive, NJ.
PETRO® LBA is available from the C.P. Hall Co. in Chicago, IL.
DOWICIL® 75 is available from Dow Chemical Co. in Midland, MI.
ALPHA-STEP® MC-38 is available from STEPAN CO. in Northfield, IL.
BURCOTERGE® PAO-35 is available from Burlington Chemical Co. in Burlington, NC.

High Pressure Lubricity Testing

The formulas were tested using Test Method No. 5, Falex High Pressure Lubricity Testing, described above. The results are shown in the following table 3. A commercially available lubricant, Conade® 2001, comparative example E, an amine based lubricant available from Ace Chemical Co., was also tested, and the results are shown in Table 4.

Table 3
Falex High Pressure Lubricity Test

	Conc. Wt-% in water	Water type	Vee Block/Pin Type	Vee Block	Test Pin	Teeth Wear (15 min. at 765 lbs)	Result
Example 1	0.50%	Well water with 19 grains of hardness per gallon	Mild steel	Very slight grooves	Very slight grooves	1	Pass
Example 1	0.40%	Well water with 19 grains of hardness per gallon	Mild steel	Very slight grooves	Very slight grooves	9	Pass
Comparative A	0.75%	Well water with 19 grains of hardness per gallon	Mild steel	Light grooves; but fails to reach 765 lbs	Light grooves; but fails to reach 765 lbs	Fails before reaching high pressure of 765 lbs	Pass
Comparative A	1.00%	Well water with 19 grains of hardness per gallon	Mild steel	Slight grooves	Slight grooves	17	Fail
Comparative B	0.40%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Severely worn	Severely worn	Fails before reaching high pressure of 765 lbs	Fail
Comparative B	0.40%	Well water with 19 grains of hardness per gallon	Mild steel	Slight black buildup with grooves	Moderate wear	39	Pass
Comparative B	0.50%	Well water with 19 grains of hardness per gallon	Mild steel	Slight black buildup with grooves	Moderate wear	15	Pass
Comparative B	0.60%	Well water with 19 grains of hardness per gallon	Mild steel	Slight black buildup with grooves	Moderate wear	25	Pass
Comparative B	0.75%	Well water with 19 grains of hardness per gallon	Mild steel	Slight black buildup with grooves	Moderate wear	38	Pass
Comparative B	0.50%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Severely worn	Severely worn	n/a	Fail
Comparative B	0.60%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Slight black buildup with grooves	Moderate wear	35	Pass
Comparative C	1.00%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Slight black buildup with grooves	Moderate wear	50	Pass
Comparative D	0.50%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Severely worn	Severely worn	n/a	Fail
Comparative D	0.75%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Slight black buildup with grooves	Severely worn	n/a	Fail
Comparative D	1.00%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Slight black buildup with grooves	Moderate wear	48	Pass

Table 4

Comp E	Conc.	Water Type	Vee Block /Pin Type	Vee Blocks	Test Pin	Teeth Wear (after 15 min./765 lbs)	Result
Conade	0.10%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Severely worn	Severely worn	n/a	Fail
Conade	0.20%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Slight black build-up	Slight wear	0	Pass
Conade	0.20%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Severely worn	Severely worn	n/a	Fail
Conade	0.30%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Slight black build-up	Slight wear	0	Pass
Conade	0.50%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Slight black build-up	Slight wear	0	Pass
Conade	0.50%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Slight black build-up	Slight wear	0	Pass
Conade	1.00%	Soft water with 0.4 grains of hardness per gallon	Mild steel	Slight black build-up	Slight wear	0	Pass

Example 1 exhibited superior high pressure lubricity as measured by the Falex High Pressure Lubricity Test Method No. 5, as compared to formulas which may be considered standards in the industry.

Slider Lubricity Testing

Slider lubricity testing was conducted according to Test Methods Nos. 3 and 4 described above. Example 1 was compared to three commercially available industrial standard lubricant formulas, comparatives E, F and G. Various test conditions were employed. The results are shown in tables 5-8, below. Slider lubricity is reported as a force reading in grams.

Table 5

Test Condition – Mild Steel on Plastic

Example	Conc. (wt-%)	Min. Force (grams)	Max. Force (grams)	Mean (grams)	Bandwidth (grams)
Comp F	0.1	18.5	19.5	19.0	1
Example 1	0.5	21.5	22.0	21.75	0.5
Comp E	0.5	17.5	18.5	18.0	1
Comp F	0.1	18.5	19.5	19.0	1

Comparative example F is MIKROGLIDE® S available from Ecolab Inc. in St. Paul, MN.

Table 6

Test Condition – Mild Steel on Stainless Steel

Example	Conc. (wt-%)	Min. Force (grams)	Max. Force (grams)	Mean (grams)	Bandwidth (grams)
Comp F	0.1	19.5	24.5	22.0	5
Example 1	0.5	23.0	30.0	26.5	7
Comp E	0.5	25.5	31.5	28.5	6
Comp F	0.1	25.5	28.0	26.75	3.5

Table 7

Test Condition – Plastic on Plastic

Example	Conc. (wt-%)	Min. Force (grams)	Max. Force (grams)	Mean (grams)	Bandwidth (grams)
Comp G	0.1	35.0	36.5	35.75	1.5
Example 1	0.5	39.0	45.0	42.0	6.0
Comp E	0.5	42.0	43.0	42.5	1.0
Comp G	0.1	36.5	38.0	37.25	1.5

Comparative example G is LUBRODRIVE® RX, commercially available from Ecolab Inc. in St. Paul, MN.

Table 8

Test Condition – Plastic on Stainless Steel

Example	Conc. (wt-%)	Min. Force (grams)	Max. Force (grams)	Mean (grams)	Bandwidth (grams)
Comp G	0.1	28.0	29.5	28.75	1.5
Comp E	0.5	33.5	35.0	34.25	1.5
Example 1	0.5	31.0	32.5	31.75	1.5
Comp G	0.1	29.0	29.0	29.5	1.0

As can be seen from tables 5-8 above, example 1 exhibits comparable slider lubricity performance to lubricants which are industry standards.

Foam Testing

Foam testing was conducted on various formulas according to Test Method No. 7 described above. Example 1 was compared to comparative examples H - L. The formulas for examples H - L are found in table 9 below. Comparative example H is essentially the same composition as in Example 1 without any foam destabilizer additive. The results of the foam testing are shown in table 10.

Table 9

Raw Material	Tradename	Function	Comp H	Comp I	Comp J	Comp K	Comp L
Oleyl ether carboxylate, 10 moles ethoxylation	EMULSOGEN® COL 100	Lubricant	10.0	----	7.50	7.5	7.5
Sulfo methyl ester	ALPHA-STEP MC-38	Lubricant	----	27.0	----	----	----
Sodium alkyl naphthalene Sulfonate; 50% active	PETRO® LBA	Coupling agent	7.0	7.0	----	10.00	10.00
C_9-C_{11} propoxylated alcohol	DEGRESSAL® SD 20	Defoamer/surfactant	----	----	----	----	----
Low foam surfactant concentrate	QUADEXX® 400	Defoamer/surfactant	----	----	10.00	----	----
Proprietary amine based gemini surfactant	SURFONYL® MD-20	Defoamer/surfactant	----	----	0.85	2.50	----
C_8-C_{10} Alkoxylated alcohol	TRITON® EF-19	Defoamer/surfactant	----	----	----	----	5.00
Chloralyl triaza azoniaadamentane	DOWICIL® 75	Biocide/preservative	0.20	0.13	0.20	0.20	0.20
Dodecyl/tetradecyloxypropyl-1,3-diaminopropane	TOMAH® DA 1618	Corrosion inhibitor	2.85	2.85	2.85	2.85	2.85
Phosphonated amine oxide	BURCOTERGE® PAO-35	Corrosion inhibitor	0.50	0.50	0.50	0.50	0.50
Dicarboxylic acid mixture	SOKALON® DCS	Corrosion inhibitor	0.85	0.85	0.85	0.85	0.85
Sodium gluconate, granular	Sodium gluconate FCC/USP	Chelates iron; rust inhibitor	2.00	2.00	2.00	2.00	2.00
Sodium laureth-13-carboxylate	SANDOPAN® LS 24N	Surfactant	----	2.85	----	----	2.85
Water, zeolite softened	Soft water		75.02	56.82	74.07	72.42	69.92
Sodium hydroxide; 50%	NaOH 50%	pH adjuster	1.58	----	1.18	1.18	1.18

SURFYNOL® MD-20 is available from Air Products and Chemicals, Inc. in Allentown, PA.
QUADEXX® 400 is available from Ecolab Inc., in Saint Paul, Minnesota
TRITON® EF 19 is available from Dow Chemical Co. in Midland, MI.

Table 10

Foam Testing Results

	Example 1	Comp H	Comp I	Comp J	Comp K	Comp L
Initial Foam	295 mls	305 mls	345 mls	295 mls	315 mls	310 mls
5 minute foam	280 mls	305 mls	330 mls	295 mls	310 mls	305 mls

Example 1 contained the alkoxylated alcohol foam destabilizer and exhibited superior performance over the same composition with no foam destabilizer. The foaming characteristics of the other types of foam destabilizers in table 9 are shown in table 10.

Example 2 and Comparative Example M

A formula having a foam suppression additive was compared to the same formula with no foam suppressor additive. The formulas were prepared as shown in table 11.

Table 11

Raw Material	Tradename	Function	Ex 2	Comp M
Oleyl ether carboxylate, 10 moles ethoxylation	EMULSOGEN® COL 100	Lubricant	8.33	10.00
C_9-C_{11} propoxylated alcohol	DEGRESSAL® SD 20	Foam suppressor	5.56	----
Sodium alkyl naphthalene Sulfonate; 50% active	PETRO® LBA	Coupling agent	7.78	7.00
Chloralyl triaza azoniaadamentane	DOWICIL® 75	Antimicrobial	0.14	0.20
Dodecyl/tetradecyloxypropyl-1,3-diaminopropane	TOMAH® D 16/18	Corrosion inhibitor	3.17	2.85
Phosphonated amine oxide	BURCOTERE® PAO 35	Corrosion inhibitor	0.56	0.50
Dicarboxylic acid mixture	SOKALON® DCS	Corrosion inhibitor	0.94	0.85
Sodium gluconate granular	Sodium gluconate FCC/USP	Chelates iron; rust inhibitor	2.22	2.00
Sodium hydrogxide, 45%	NaOH, 50%	pH adjuster	1.31	1.58
Water, zeolite softened			69.99	75.02

Foam testing was conducted according to Test Method No. 8. The results are shown in table 12.

Table 12
Short Track Conveyor Foam Testing

Name	Example 2	Comparative example M
Length of test	3.5 hours	3.5 hours
Concentration	0.5% in well water	0.5% in well water
Appearance of track	small bubbles (most about 2-5 mm in diameter, sparsely dispersed (about 5% of track surface had foam)	medium bubbles (5-8 mm, covering about 30-40% of track surface)
Appearance of drip pan	mostly clear water, less than 5% of water's surface had bubbles	100% of liquid in drip pan covered in foam, which was building to 1-2 inches in some areas
Other observations	some foam was clinging beneath the track in a few places	considerable foam was clinging beneath the track in several places. Foam was clinging to other surfaces (notably the axle of the neighboring short track, which is 90 degrees from horizontal)
Foam level (bottom line)	low to moderate	high

As can be seen from the data, Example 1, having a foam suppressor additive included with the lubricant, exhibited considerably less foaming.

Examples 3 and 4 and Comparative Example E, Conade® 2001

Examples 3 and 4 were prepared for milk coagulation testing. Example 3 is essentially the same formulation as example 1, while example 4 has an increased level of ether carboxylate lubricant. The formulas are shown in Table 13.

Table 13

Raw Material	Tradename	Function	Ex 3	Ex 4
Oleyl ether carboxylate, 10 moles ethoxylation	EMULSOGEN® COL 100	Lubricant	7.50	9.00
Sodium alkyl naphthalene Sulfonate; 50% active	PETRO® LBA	Coupling agent	7.00	7.00
C_9-C_{11} Alkoxylated alcohol	DEGRESSAL® SD 20	Defoamer/surfactant	3.50	5.00
Chloralyl triaza azoniaadamentane	DOWICIL® 75	Biocide/preservative	0.20	0.130
Dodecyl/tetradecyloxypropyl-1,3-diaminopropane	TOMAH® DA 1618	Corrosion inhibitor	2.85	2.85
Phosphated amine oxide	BURCOTERGE® PAO-35	Corrosion inhibitor	0.50	0.50
Dicarboxylic acid mixture	SOKALON® DCS	Corrosion inhibitor	0.85	0.85
Sodium gluconate, granular	Sodium gluconate FCC/USP	Chelates iron; rust inhibitor	2.00	2.00
Water, zeolite softened	Soft water	Solution base	72.92	73.41
Sodium hydroxide; 50%	NaOH 50%	pH adjuster	1.18	0.76

After mixing, example 3 was slightly blue with a very slight haze and example 4 was initially opaque (5 minutes). Both solutions completely cleared overnight.

Milk Coagulation Testing

Milk coagulation testing for examples 3 and 4 was conducted according to Test Method No. 1, described above, and compared to a commercially available amine based lubricant, CONADE® 2001, comparative example E, available from Ace Chemical Co.

Examples 3 and 4 both exhibited no ring of precipitate from coagulation, but a streak was visible where the solution had been after pouring it off the panel, while CONADE® 2001, comparative example E, produced a pronounced ring of precipitate from coagulation and after pouring the solution off the panel, also had streaks visible where the solution had been.

Milk coagulation testing was conducted according to Test Method No. 2, described above. Example 3 exhibited a solution which was clearer on top, but whiter and more opaque at the bottom. No precipitates were visible on the bottom of the

beaker. CONADE® 2001, comparative example E, was also clearer at the top, more opaque and whiter on the bottom, and had a definite precipitate on the bottom of the beaker. The samples were again observed the following day. There was no change in results.

Corrosion Testing

Samples were prepared and corrosion testing was conducted according to Test Method No. 6, described above. After 30 minutes, Example 3 exhibited slight orange corrosion marks near the liquid-air interface on the front and back of the panel and example 4 exhibited three small corrosion marks on the front of the panel at the liquid-air interface line. Comparative example E exhibited no corrosion.

After 2 ½ hours, example 3 exhibited a 1 inch line of corrosion on the front and back of the panel at liquid-air interface line and example 4 exhibited 1/8" corrosion on each edge on the back of the panel only along the liquid-air interface line. Three very small corrosion marks were noted on the front of the panel at the liquid-air interface line. Again, comparative E exhibited no corrosion.

After 18 ½ hours, example 3 exhibited corrosion at and a little bit above the liquid line on the back of the panel (~2" on the front of the pane and ~1" on the back of the panel), and one small mark below the liquid-air interface line on the front and example 4 exhibited a small amount of corrosion at the liquid-air interface line on the front and back edges, each ~1/8". Comparative example E exhibited no corrosion.

A panel was placed in water from the Golden Guernsey Dairy of Waukesha, WI (19 grain per gallon hardness as calcium carbonate or 325 parts per million as calcium carbonate) for comparison to the above tests. Signs of corrosion were visible on the panel within fifteen minutes. After 18½ hours, a strong line of corrosion was visible at the liquid-air interface. All (100%) of the panel which had been immersed in the solution was corroded and a layer of rust coated the beaker bottom.

While comparative example E, Conade® 2001, a lubricant which is an industry standard, exhibited slightly better corrosion resistance, examples 3 and 4 exhibited acceptable levels of corrosion resistance.

The above disclosure is intended for illustrative purposes only and is not exhaustive. The embodiments described therein will suggest many variations and alternatives to one of ordinary skill in this art. All these alternatives and variations are intended to be included within the scope of the attached claims. Those familiar with the

art may recognize other equivalents to the specific embodiments described herein which equivalents are also intended to be encompassed by the claims attached hereto.